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4-Hydroxy-5-(2-methoxyphenoxy)-2,2′-bipyrimidin-6(5*H*)-one dihydrate

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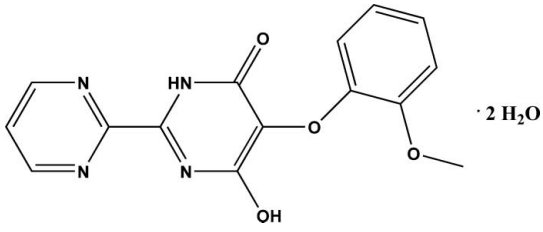
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.041; wR factor = 0.115; data-to-parameter ratio = 12.2.

The title compound, $C_{15}H_{12}N_4O_4\cdot 2H_2O$, crystallizes with two independent water molecules in the asymmetric unit. The dihedral angles between the mean planes of the benzene and pyrimidine rings and that of the pyrimidin-4-one ring are 85.1 (9) and 82.1 (1)°, respectively. The mean plane of the pyrimidine ring is twisted by 12.8 (8)° from that of the pyrimidin-4-one ring. The dihedral angles between the benzene ring and the mean planes of the pyrimidine and pyrimidin-4-one rings are 85.1 (9) and 82.1 (1)°, respectively.In the crystal, N–H···O, O—H···N and O—H···O hydrogen bonds involving both water molecules are present; these link the molecules into a two-dimensional network parallel to (010). In addition, weak C—H···π and π–π [centroid–centroid distance = 3.6183 (8) Å] interactions occur.

Related literature

For substituted pyrimidine-2,4-diones as good reversible inhibitors of thymidine phosphorylase, see: Baker & Rzeszotarki (1967). For the use of 2,2′-bipyrimidine as a ligand in inorganic and organometallic chemistry, see: Hunziker & Ludi (1977); Fabrice *et al.* (2008). For related structures, see: El-Brollosy *et al.* (2012); Fun *et al.* (2009); Kaur *et al.* (2013); Ren *et al.* (2011); Trilleras *et al.* (2009).



Experimental

Crystal data

$C_{15}H_{12}N_4O_4\cdot 2H_2O$
M_r = 348.32
Monoclinic, $P2_1/c$
a = 9.5817 (3) Å
b = 13.9439 (3) Å
c = 12.4804 (4) Å
β = 109.832 (3)°
V = 1568.55 (8) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 0.99 mm^{-1}
T = 173 K
0.45 × 0.32 × 0.24 mm

Data collection

Agilent Xcalibur (Eos, Gemini) diffractometer
Absorption correction: multi-scan (*CrysAlis PRO* and *CrysAlis RED*; Agilent, 2012)
T_{min} = 0.876, T_{max} = 1.000
9840 measured reflections
3070 independent reflections
2789 reflections with $I > 2\sigma(I)$
R_{int} = 0.040

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.115
S = 1.03
3070 reflections
252 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

*Cg*3 is the centroid of the C9–C14 ring.

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O2W^{i}	0.91 (3)	1.67 (3)	2.5651 (15)	172 (2)
N4—H4···O1^{ii}	0.88 (2)	2.08 (2)	2.9313 (15)	163.6 (18)
O1W—H1WA···O4^{iii}	0.87 (2)	2.05 (2)	2.8917 (16)	163 (2)
O1W—H1WB···N2^{iv}	0.89 (3)	2.01 (3)	2.8823 (16)	167 (2)
O2W—H2WA···O4	0.86 (2)	1.93 (3)	2.7803 (15)	172 (2)
O2W—H2WB···O1W^{v}	0.91 (3)	1.80 (3)	2.7050 (17)	173 (2)
C4—H4A···$Cg3^{vi}$	0.95	2.82	3.4083 (16)	121

Symmetry codes: (i) $x+1, -y+\frac{1}{2}, z+\frac{1}{2}$; (ii) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (iii) $-x+1, -y+1, -z+1$; (iv) $-x+2, -y+1, -z+1$; (v) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$; (vi) $x, -y-\frac{1}{2}, z-\frac{3}{2}$.

Data collection: *CrysAlis PRO* (Agilent, 2012); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis RED* (Agilent, 2012); program(s) used to solve structure: *SUPERFLIP* (Palatinus & Chapuis, 2007); program(s) used to refine structure: *SHELXL2012* (Sheldrick, 2008); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT6940).

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supplementary materials

Acta Cryst. (2013). E**69**, o1707–o1708 [doi:10.1107/S1600536813028900]

4-Hydroxy-5-(2-methoxyphenoxy)-2,2′-bipyrimidin-6(5*H*)-one dihydrate

Thammarse S. Yamuna, Jerry P. Jasinski, Brian J. Anderson, H.S. Yathirajan and Manpreet Kaur

1. Comment

The title compound, $C_{15}H_{12}N_4O_4$. 2 H_2O, is an intermediate for the synthesis of bosentan. Pyrimidinediones are a class of chemical compounds characterized by a pyrimidine ring substituted with two carbonyl groups. A number of substituted pyrimidine-2,4-diones were synthesized and intensively studied as good reversible inhibitors of thymidine phosphorylase (Baker & Rzeszotarki,1967). 2,2′-Bipyrimidine has been used as a ligand in inorganic and organometallic chemistry (Hunziker & Ludi, 1977; Fabrice *et al.*, 2008). The crystal structures of some related compounds are: four 7-aryl-substituted pyrido[2,3-d]pyrimidine -2,4(1H,3H)-diones: similar molecular structures but different crystal structures (Trilleras *et al.*, 2009), diammonium 1,1′,3,3′-tetra methyl-2,2′,4,4′,6,6′-hexaoxoperhydro-5,5′-bipyrimidine-5,5′-diide monohydrate (Fun *et al.*, 2009), 4,6-dichloro-5-(2-methoxy phenoxy)-2,2′-bipyrimidine (Ren *et al.*, 2011), 6-(3,5-di-methyl benzyl)-5-ethyl-1-[(3-phenylpropoxy) methyl]-1,2,3,4-tetrahydro pyrimidine-2,4-dione (El-Brollosy *et al.*, 2012), 4-tert-butyl- N-[6-(2-hydroxyethoxy)-5-(2-methoxyphenoxy)-2-(pyrimidin-2-yl)pyrimidin -4-yl]benzene-1-sulfonamide monohydrate (Kaur *et al.*, 2013), have been reported. In view of the importance of the title compound this paper reports its crystal structure.

The title compound crystallizes with two independent water molecules in the asymmetric unit (Fig. 1). In the molecule, the dihedral angle between the mean planes of the phenyl ring and pyrimidine ring and the pyrimidinyl-4-one is 85.1 (9)° and 82.1 (1)°, respectively. The mean plane of the pyrimidine ring is twisted by 12.8 (8)° from that of the pyrimidinyl-4-one ring. In addition, the hydroxy group and methoxy group are twisted from the pyrimidinyl-4-one and phenyl ring by -177.6 (8)° (H3/O3/C8/C7) and -173.3 (4)° (C9/C14/O1/C15), respectively. In the crystal, O—H···N and O—H···O hydrogen bonds involving both water molecules are present. N—H···O hydrogen bonds between molecules help strengthen the crystal lattice (Table. 1). In addition, weak C4—H4A···Cg3 and Cg1–Cg2 π–π intermolecular interactions are observed and contribute to crystal packing (Cg1–Cg2 = 3.6183 (8)Å; Cg1 = N1/C1/N2/C2/C3/C4; $Cg2^{i}$ = N3/C5/N4/C6/C7/C8; symmetry operator (i) -x,1-y,1-z). The hydrogen bonds link the molecules into a 2D network parallel to (0 1 0) (Fig. 2).

2. Experimental

The title compound was obtained as a gift sample from Cadila Pharmaceuticals, Ahmedabad. The title compound (0.5 g) was dissolved in 10 ml of a mixture of dimethylsulphoxide and dimethylformamide (1:1) and stirred for 30 minutes on magnetic stirrer at 308 K. X-ray quality crystals of the title compound were formed after few days (m. p.: 536–540 K).

3. Refinement

H3, H4, H1WA, H1WB, H2WA and H2WB were located by a difference map and refined isotropically. All of the remaining H atoms were placed in their calculated positions and then refined using the riding model with Atom—H lengths of 0.95Å (CH) or 0.98Å (CH_3). Isotropic displacement parameters for these atoms were set to 1.2 (CH) or 1.5

(CH_3) times U_{eq} of the parent atom. The methyl group was refined as a rotating group.

Computing details

Data collection: *CrysAlis PRO* (Agilent, 2012); cell refinement: *CrysAlis PRO* (Agilent, 2012); data reduction: *CrysAlis RED* (Agilent, 2012); program(s) used to solve structure: SUPERFLIP (Palatinus & Chapuis, 2007); program(s) used to refine structure: *SHELXL2012* (Sheldrick, 2008); molecular graphics: OLEX2 (Dolomanov *et al.*, 2009); software used to prepare material for publication: OLEX2 (Dolomanov *et al.*, 2009).

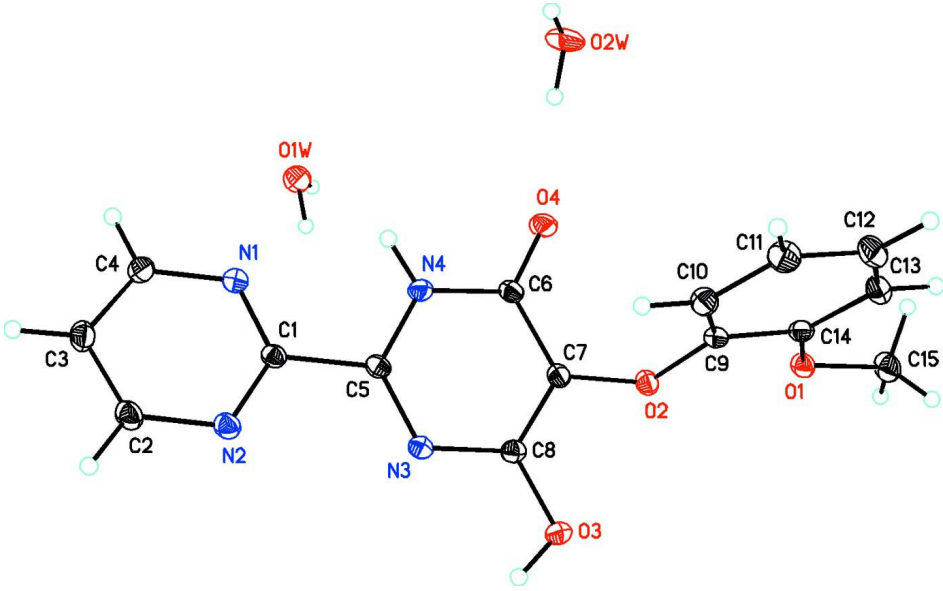

Figure 1

ORTEP drawing of (I) ($C_{15}H_{12}N_4O_4$. 2 H_2O) showing the labeling scheme with 30% probability displacement ellipsoids.

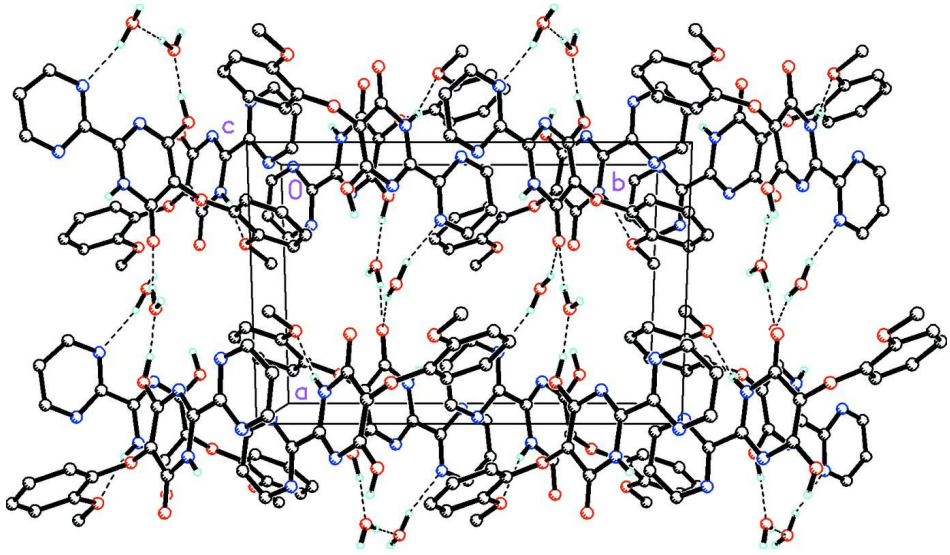

Figure 2

Molecular packing for (I) viewed along the *C* axis. Dashed lines indicate O—H···N and O—H···O hydrogen bonds involving both water molecules and N—H···O hydrogen bonds between the molecules. All of these interactions directly link the molecules into a 2D network along (0 1 0). H atoms not involved in hydrogen bonding have been removed for clarity.

4-Hydroxy-5-(2-methoxyphenoxy)-2,2′-bipyrimidin-6(5*H*)-one dihydrate

Crystal data

$C_{15}H_{12}N_4O_4·2H_2O$
$M_r = 348.32$
Monoclinic, $P2_1/c$
$a = 9.5817$ (3) Å
$b = 13.9439$ (3) Å
$c = 12.4804$ (4) Å
$\beta = 109.832$ (3)°
$V = 1568.55$ (8) Å^3
$Z = 4$
$F(000) = 728$
$D_x = 1.475$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 4761 reflections
$\theta = 3.2$–$72.3°$
$\mu = 0.99$ mm^{-1}
$T = 173$ K
Irregular, colourless
0.45 × 0.32 × 0.24 mm

Data collection

Agilent Xcalibur (Eos, Gemini) diffractometer
Radiation source: Enhance (Cu) X-ray Source
Detector resolution: 16.0416 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO* and *CrysAlis RED*; Agilent, 2012)
$T_{min} = 0.876$, $T_{max} = 1.000$
9840 measured reflections
3070 independent reflections
2789 reflections with $I > 2\sigma(I)$
$R_{int} = 0.040$
$\theta_{max} = 72.5°$, $\theta_{min} = 4.9°$
$h = -11 \rightarrow 11$
$k = -17 \rightarrow 13$
$l = -15 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.115$
$S = 1.03$
3070 reflections
252 parameters
0 restraints

Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0697P)^2 + 0.4106P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}
Extinction correction: *SHELXL2012* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0118 (8)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.66810 (10)	0.07758 (7)	0.72585 (8)	0.0234 (2)
O2	0.83857 (10)	0.17796 (7)	0.64227 (8)	0.0218 (2)
O3	1.14014 (11)	0.20971 (7)	0.71581 (8)	0.0261 (3)
H3	1.235 (3)	0.2265 (17)	0.727 (2)	0.059 (7)*
O4	0.67224 (10)	0.27899 (8)	0.44713 (8)	0.0284 (3)
N1	0.99634 (13)	0.47988 (8)	0.33822 (10)	0.0233 (3)
N2	1.24321 (12)	0.42848 (8)	0.43964 (10)	0.0228 (3)
N3	1.12057 (12)	0.31273 (8)	0.56724 (9)	0.0199 (3)
N4	0.88527 (12)	0.34237 (8)	0.43264 (10)	0.0214 (3)
C1	1.09611 (14)	0.42507 (9)	0.41329 (11)	0.0195 (3)
C2	1.29246 (15)	0.49577 (11)	0.38523 (12)	0.0268 (3)
H2	1.3965	0.5022	0.4023	0.032*
C3	1.19918 (17)	0.55649 (10)	0.30517 (12)	0.0284 (3)
H3A	1.2366	0.6040	0.2675	0.034*
C4	1.04900 (16)	0.54475 (10)	0.28272 (12)	0.0261 (3)
H4A	0.9812	0.5838	0.2264	0.031*
C5	1.03430 (14)	0.35477 (9)	0.47561 (11)	0.0189 (3)
C6	0.80944 (14)	0.28493 (9)	0.48529 (11)	0.0205 (3)
C7	0.90348 (14)	0.23650 (9)	0.58376 (11)	0.0196 (3)
C8	1.05471 (14)	0.25246 (9)	0.62200 (11)	0.0198 (3)
C9	0.79005 (14)	0.08924 (9)	0.59160 (11)	0.0202 (3)
C10	0.82984 (16)	0.05252 (10)	0.50337 (12)	0.0255 (3)
H10	0.8894	0.0895	0.4715	0.031*
C11	0.78253 (18)	−0.03891 (11)	0.46092 (13)	0.0314 (3)
H11	0.8091	−0.0640	0.3997	0.038*
C12	0.69761 (19)	−0.09249 (11)	0.50761 (14)	0.0341 (4)
H12	0.6664	−0.1550	0.4792	0.041*
C13	0.65690 (17)	−0.05569 (11)	0.59662 (13)	0.0294 (3)
H13	0.5980	−0.0931	0.6285	0.035*
C14	0.70226 (14)	0.03557 (10)	0.63884 (11)	0.0211 (3)
C15	0.59236 (18)	0.01817 (12)	0.78172 (13)	0.0329 (4)
H15A	0.5822	0.0526	0.8472	0.049*
H15B	0.6492	−0.0409	0.8080	0.049*
H15C	0.4937	0.0021	0.7283	0.049*

O1W	0.50931 (13)	0.68034 (8)	0.41460 (10)	0.0335 (3)
O2W	0.41708 (12)	0.25759 (10)	0.25780 (10)	0.0385 (3)
H2WA	0.493 (3)	0.2699 (16)	0.316 (2)	0.049 (6)*
H2WB	0.445 (3)	0.2361 (16)	0.199 (2)	0.055 (6)*
H4	0.836 (2)	0.3704 (14)	0.3675 (18)	0.037 (5)*
H1WA	0.460 (3)	0.7048 (16)	0.455 (2)	0.050 (6)*
H1WB	0.592 (3)	0.6557 (17)	0.464 (2)	0.058 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0213 (5)	0.0276 (5)	0.0228 (5)	−0.0045 (4)	0.0095 (4)	−0.0001 (4)
O2	0.0242 (5)	0.0226 (5)	0.0198 (5)	−0.0055 (3)	0.0091 (4)	−0.0022 (3)
O3	0.0201 (5)	0.0306 (5)	0.0234 (5)	−0.0006 (4)	0.0017 (4)	0.0072 (4)
O4	0.0149 (5)	0.0421 (6)	0.0260 (5)	−0.0032 (4)	0.0041 (4)	0.0052 (4)
N1	0.0211 (6)	0.0246 (6)	0.0225 (6)	−0.0014 (4)	0.0051 (4)	0.0012 (4)
N2	0.0186 (6)	0.0276 (6)	0.0228 (6)	−0.0015 (4)	0.0080 (4)	−0.0011 (4)
N3	0.0162 (5)	0.0217 (6)	0.0207 (6)	−0.0004 (4)	0.0049 (4)	−0.0004 (4)
N4	0.0160 (5)	0.0271 (6)	0.0195 (6)	0.0000 (4)	0.0036 (4)	0.0034 (4)
C1	0.0191 (6)	0.0214 (6)	0.0181 (6)	−0.0020 (5)	0.0062 (5)	−0.0034 (5)
C2	0.0213 (7)	0.0352 (8)	0.0266 (7)	−0.0055 (5)	0.0115 (6)	−0.0021 (6)
C3	0.0341 (8)	0.0287 (7)	0.0249 (7)	−0.0079 (6)	0.0135 (6)	−0.0001 (5)
C4	0.0294 (7)	0.0245 (7)	0.0220 (7)	−0.0022 (5)	0.0056 (5)	0.0015 (5)
C5	0.0163 (6)	0.0202 (6)	0.0204 (6)	−0.0004 (4)	0.0064 (5)	−0.0028 (5)
C6	0.0169 (6)	0.0250 (7)	0.0196 (6)	−0.0029 (5)	0.0061 (5)	−0.0025 (5)
C7	0.0199 (7)	0.0204 (6)	0.0193 (6)	−0.0031 (5)	0.0076 (5)	−0.0016 (5)
C8	0.0196 (6)	0.0197 (6)	0.0188 (6)	0.0010 (5)	0.0047 (5)	−0.0012 (5)
C9	0.0176 (6)	0.0214 (6)	0.0187 (6)	−0.0010 (5)	0.0022 (5)	0.0001 (5)
C10	0.0268 (7)	0.0274 (7)	0.0236 (7)	−0.0028 (5)	0.0101 (5)	−0.0012 (5)
C11	0.0385 (8)	0.0304 (8)	0.0259 (7)	−0.0003 (6)	0.0117 (6)	−0.0065 (6)
C12	0.0416 (9)	0.0246 (7)	0.0333 (8)	−0.0079 (6)	0.0090 (7)	−0.0081 (6)
C13	0.0302 (7)	0.0274 (7)	0.0297 (7)	−0.0087 (6)	0.0090 (6)	−0.0007 (6)
C14	0.0176 (6)	0.0258 (7)	0.0179 (6)	−0.0007 (5)	0.0034 (5)	0.0008 (5)
C15	0.0335 (8)	0.0399 (8)	0.0299 (8)	−0.0119 (6)	0.0167 (6)	0.0000 (6)
O1W	0.0237 (6)	0.0415 (6)	0.0361 (6)	0.0028 (4)	0.0114 (5)	0.0038 (5)
O2W	0.0189 (5)	0.0652 (8)	0.0275 (6)	−0.0042 (5)	0.0028 (5)	−0.0095 (5)

Geometric parameters (Å, º)

O1—C14	1.3676 (16)	C4—H4A	0.9500
O1—C15	1.4292 (16)	C6—C7	1.4237 (19)
O2—C7	1.3765 (15)	C7—C8	1.3812 (18)
O2—C9	1.3952 (16)	C9—C10	1.3802 (19)
O3—H3	0.91 (3)	C9—C14	1.3970 (18)
O3—C8	1.3212 (16)	C10—H10	0.9500
O4—C6	1.2398 (16)	C10—C11	1.396 (2)
N1—C1	1.3297 (18)	C11—H11	0.9500
N1—C4	1.3378 (18)	C11—C12	1.372 (2)
N2—C1	1.3351 (17)	C12—H12	0.9500
N2—C2	1.3347 (18)	C12—C13	1.394 (2)

N3—C5	1.3013 (17)	C13—H13	0.9500
N3—C8	1.3653 (17)	C13—C14	1.390 (2)
N4—C5	1.3554 (16)	C15—H15A	0.9800
N4—C6	1.3874 (17)	C15—H15B	0.9800
N4—H4	0.88 (2)	C15—H15C	0.9800
C1—C5	1.4930 (17)	O1W—H1WA	0.87 (2)
C2—H2	0.9500	O1W—H1WB	0.89 (3)
C2—C3	1.381 (2)	O2W—H2WA	0.86 (2)
C3—H3A	0.9500	O2W—H2WB	0.91 (3)
C3—C4	1.379 (2)		

C14—O1—C15	115.91 (11)	O3—C8—N3	117.94 (12)
C7—O2—C9	115.24 (10)	O3—C8—C7	119.70 (12)
C8—O3—H3	108.2 (15)	N3—C8—C7	122.35 (12)
C1—N1—C4	116.50 (12)	O2—C9—C14	116.07 (11)
C2—N2—C1	115.19 (12)	C10—C9—O2	123.42 (12)
C5—N3—C8	116.97 (11)	C10—C9—C14	120.45 (12)
C5—N4—C6	122.48 (11)	C9—C10—H10	120.0
C5—N4—H4	118.2 (13)	C9—C10—C11	119.98 (13)
C6—N4—H4	119.3 (13)	C11—C10—H10	120.0
N1—C1—N2	126.84 (12)	C10—C11—H11	120.0
N1—C1—C5	115.28 (11)	C12—C11—C10	119.95 (13)
N2—C1—C5	117.86 (11)	C12—C11—H11	120.0
N2—C2—H2	118.5	C11—C12—H12	119.8
N2—C2—C3	123.04 (13)	C11—C12—C13	120.31 (14)
C3—C2—H2	118.5	C13—C12—H12	119.8
C2—C3—H3A	121.7	C12—C13—H13	119.9
C4—C3—C2	116.66 (13)	C14—C13—C12	120.22 (13)
C4—C3—H3A	121.7	C14—C13—H13	119.9
N1—C4—C3	121.72 (13)	O1—C14—C9	116.50 (12)
N1—C4—H4A	119.1	O1—C14—C13	124.41 (12)
C3—C4—H4A	119.1	C13—C14—C9	119.09 (12)
N3—C5—N4	124.06 (12)	O1—C15—H15A	109.5
N3—C5—C1	120.51 (11)	O1—C15—H15B	109.5
N4—C5—C1	115.37 (11)	O1—C15—H15C	109.5
O4—C6—N4	120.94 (12)	H15A—C15—H15B	109.5
O4—C6—C7	125.30 (12)	H15A—C15—H15C	109.5
N4—C6—C7	113.76 (11)	H15B—C15—H15C	109.5
O2—C7—C6	118.15 (11)	H1WA—O1W—H1WB	107 (2)
O2—C7—C8	121.47 (12)	H2WA—O2W—H2WB	111 (2)
C8—C7—C6	120.29 (12)		

O2—C7—C8—O3	1.07 (19)	C5—N4—C6—O4	176.19 (12)
O2—C7—C8—N3	−177.80 (11)	C5—N4—C6—C7	−3.53 (18)
O2—C9—C10—C11	176.86 (13)	C6—N4—C5—N3	2.0 (2)
O2—C9—C14—O1	2.85 (17)	C6—N4—C5—C1	−175.25 (11)
O2—C9—C14—C13	−176.50 (12)	C6—C7—C8—O3	177.58 (12)
O4—C6—C7—O2	0.1 (2)	C6—C7—C8—N3	−1.29 (19)
O4—C6—C7—C8	−176.55 (13)	C7—O2—C9—C10	13.23 (18)

N1—C1—C5—N3	−164.82 (12)	C7—O2—C9—C14	−169.66 (11)
N1—C1—C5—N4	12.53 (16)	C8—N3—C5—N4	0.20 (19)
N2—C1—C5—N3	13.60 (18)	C8—N3—C5—C1	177.32 (11)
N2—C1—C5—N4	−169.04 (11)	C9—O2—C7—C6	76.38 (14)
N2—C2—C3—C4	0.0 (2)	C9—O2—C7—C8	−107.04 (13)
N4—C6—C7—O2	179.77 (10)	C9—C10—C11—C12	−0.6 (2)
N4—C6—C7—C8	3.15 (18)	C10—C9—C14—O1	−179.95 (12)
C1—N1—C4—C3	−2.3 (2)	C10—C9—C14—C13	0.7 (2)
C1—N2—C2—C3	−1.5 (2)	C10—C11—C12—C13	0.7 (2)
C2—N2—C1—N1	1.32 (19)	C11—C12—C13—C14	−0.1 (2)
C2—N2—C1—C5	−176.90 (11)	C12—C13—C14—O1	−179.89 (13)
C2—C3—C4—N1	2.0 (2)	C12—C13—C14—C9	−0.6 (2)
C4—N1—C1—N2	0.5 (2)	C14—C9—C10—C11	−0.1 (2)
C4—N1—C1—C5	178.78 (11)	C15—O1—C14—C9	−173.34 (12)
C5—N3—C8—O3	−179.40 (11)	C15—O1—C14—C13	5.96 (19)
C5—N3—C8—C7	−0.51 (18)		

Hydrogen-bond geometry (Å, º)

Cg3 is the centroid of the C9–C14 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O2*W*^{i}	0.91 (3)	1.67 (3)	2.5651 (15)	172 (2)
N4—H4···O1^{ii}	0.88 (2)	2.08 (2)	2.9313 (15)	163.6 (18)
O1*W*—H1*WA*···O4^{iii}	0.87 (2)	2.05 (2)	2.8917 (16)	163 (2)
O1*W*—H1*WB*···N2^{iv}	0.89 (3)	2.01 (3)	2.8823 (16)	167 (2)
O2*W*—H2*WA*···O4	0.86 (2)	1.93 (3)	2.7803 (15)	172 (2)
O2*W*—H2*WB*···O1*W*^{v}	0.91 (3)	1.80 (3)	2.7050 (17)	173 (2)
C4—H4*A*···*Cg*3^{vi}	0.95	2.82	3.4083 (16)	121

Symmetry codes: (i) *x*+1, −*y*+1/2, *z*+1/2; (ii) *x*, −*y*+1/2, *z*−1/2; (iii) −*x*+1, −*y*+1, −*z*+1; (iv) −*x*+2, −*y*+1, −*z*+1; (v) −*x*+1, *y*−1/2, −*z*+1/2; (vi) *x*, −*y*−1/2, *z*−3/2.